PB90-239971

Audit Materials for Semivolatile Organic Measurements during Hazardous Waste Trial Burns

Research Triangle Inst., Research Triangle Park, NC

Prepared for:

Environmental Protection Agency, Research Triangle Park, NC

Apr 90

U.S. Department of Commerce
National Technical Information Service
NTIS

PB90-239971

EPA/600/3-90/033
April 1990

AUDIT MATERIALS FOR SEMIVOLATILE ORGANIC MEASUREMENTS DURING HAZARDOUS WASTE TRIAL BURNS

by

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EPA Contracts: 68-02-3992, 68-02-4125, and 68-02-4550

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ATMOSPHERIC RESEARCH AND EXPOSURE ASSESSMENT LABORATORY
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TECHNICAL REPORT DATA
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1. REPORT NO. EPA/600/3-90/033	2.
3. PB90-239971	
4. TITLE AND SUBTITLE Audit Materials for Semivolatile Organic Measurements During Hazardous Waste Trial Burns	5. REPORT DATE April 1990
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) J.R. Albritton, R.S. Wright, W.C. Eaton, and R.K.M. Jayanty	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Research Triangle Institute Research Triangle Park, NC	10. PROGRAM ELEMENT NO.
	11. CONTRACT/GRANT NO. 68-02-3942 68-02-4550 68-02-4125
12. SPONSORING AGENCY NAME AND ADDRESS U.S. Environmental Protection Agency AREAL/Quality Assurance Division (MD-77A) Research Triangle Park, NC 27711	13. TYPE OF REPORT AND PERIOD COVERED Project Report and Summary
	14. SPONSORING AGENCY CODE EPA/600/09
15. SUPPLEMENTARY NOTES	

16. ABSTRACT

Two new performance audit materials utilizing different sorbents have been developed to assess the overall accuracy and precision of the sampling, desorption, and analysis of semivolatile organic compounds by EPA, SW 846 Method 0010 (i.e., the Modified Method 5 sampling train). The first audit material is an XAD-2 cartridge. It is prepared by spiking known quantities of organic compounds onto XAD-2 resin contained in glass cartridges. The test compounds are recovered and analyzed by using Method 0010 laboratory procedures. Earlier studies at RTI showed recovery efficiencies to be between 88 and 98 percent. Test compounds spiked onto XAD-2 cartridges show little change (± 6 percent) in the amount loaded when stored under refrigeration over an 8-month period. The second audit material is a Carbotrap™ cartridge. It is prepared by spiking known quantities of test compounds onto Carbotrap adsorbent material contained in stainless steel cartridges. The test compounds are thermally desorbed from the cartridges onto an operating Method 0010 unit. The test compounds are then recovered by using Method 0010 laboratory procedures. Earlier studies at RTI showed recovery efficiencies to be between 80 and 108 percent. Five of the six test compounds spiked onto Carbotrap cartridges showed little change (+ 7 percent) in the amount loaded when stored at room temperatures over a two-month period. Nitrobenzene does not appear to be stable on Carbotrap audit cartridges. An interlaboratory study to quantitate the accuracy and precision of the audit materials was conducted with five

17. KEY WORDS AND DOCUMENT ANALYSIS (CONTINUED ON ATTACHED SHEET)

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Modified Method 5 Semivolatiles XAD-2 Carbotrap Sw-846 Method 0010		

18. DISTRIBUTION STATEMENT RELEASE TO PUBLIC	19. SECURITY CLASS *(This Report)* Unclassified	21. NO. OF PAGES 40
	20. SECURITY CLASS *(This page)* Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

cooperating laboratories. The overall mean bias percentage for the interlaboratory study was -29 percent. The mean bias percentages for individual laboratories ranged from -5 to -60 percent. The between-laboratories variabilities (i.e., the relative standard deviations of mean measured loadings for the five laboratories) ranged from 10 to 68 percent with a mean value of 42 percent. The within-laboratory variabilities (i.e., the relative standard deviations of triplicate measurements within each laboratory) ranged from 0 to 94 percent with a mean value of 22 percent. Mean within-laboratory variabilities for individual laboratories ranged from 8 to 49 percent. These between-laboratories and within-laboratory variabilities generally agree with corresponding values obtained in an interlaboratory comparison study of methods for volatile and semivolatile compounds that was sponsored by EPA-Las Vegas.[9]

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems for air, implementation of agency-wide quality assurance programs for air pollution measurement systems, and supplying technical support to other groups in the Agency including the Office of Air and Radiation, the Office of Toxic Substances, and the Office of Solid Waste.

Method 0010 of SW 846 is the required test method used to show Destruction and Removal Efficiency (DRE) of Principle Organic Hazardous Constituents (POHCs) of semi-volatile (boiling point above 130 °C) organic compounds. This method requires extraction and evaporation concentrating of the semi-volatile organics. This procedure, if not done properly, can result in significant loss of POHCs and, therefore, it is important to have an audit material to test the performance of laboratories making these important measurements. Two approaches for the audit material are described in this report: one which tests both the sample train recovery and analytical recovery and one which tests the analytical performance only.

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ABSTRACT

Two new performance audit materials utilizing different sorbents have been developed to assess the overall accuracy and precision of the sampling, desorption, and analysis of semivolatile organic compounds by EPA, SW 846 Method 0010 (i.e., the Modified Method 5 sampling train). The first audit material is an XAD-2 cartridge. It is prepared by spiking known quantities of organic compounds onto XAD-2 resin contained in glass cartridges. The test compounds are recovered and analyzed by using Method 0010 laboratory procedures. Earlier studies at RTI showed recovery efficiencies to be between 88 and 98 percent. Test compounds spiked onto XAD-2 cartridges show little change (± 6 percent) in the amount loaded when stored under refrigeration over an 8-month period. The second audit material is a Carbotrap™ cartridge. It is prepared by spiking known quantities of test compounds onto Carbotrap adsorbent material contained in stainless steel cartridges. The test compounds are thermally desorbed from the cartridges onto an operating Method 0010 unit. The test compounds are then recovered by using Method 0010 laboratory procedures. Earlier studies at RTI showed recovery efficiencies to be between 80 and 108 percent. Five of the six test compounds spiked onto Carbotrap cartridges showed little change (±7 percent) in the amount loaded when stored at room temperatures over a two-month period. Nitrobenzene does not appear to be stable on Carbotrap audit cartridges. An interlaboratory study to quantitate the accuracy and precision of the audit materials was conducted with five cooperating laboratories. The overall mean bias percentage for the interlaboratory study was -29 percent. The mean bias percentages for individual laboratories ranged from -5 to -60 percent. The between-laboratories variabilities (i.e., the relative standard deviations of mean measured loadings for the five laboratories) ranged from 10 to 68 percent

with a mean value of 42 percent. The within-laboratory variabilities (i.e., the relative standard deviations of triplicate measurements within each laboratory) ranged from 0 to 94 percent with a mean value of 22 percent. Mean within-laboratory variabilities for individual laboratories ranged from 8 to 49 percent. These between-laboratories and within-laboratory variabilities generally agree with corresponding values obtained in an interlaboratory comparison study of methods for volatile and semivolatile compounds that was sponsored by EPA-Las Vegas.[9]

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SECTION 1

INTRODUCTION

Operators of hazardous waste incinerators are currently using Method 0010 to monitor stack emissions during hazardous waste trial burns[1]. The sampling train consists of a series of traps and impingers that collect particulates and gaseous semivolatile organics. An adsorbent material, XAD-2 resin, is used to adsorb organic vapors with boiling points greater than 100 °C. The overall accuracy of the process of trapping, desorbing, and analyzing these organic vapors by Method 0010 can be assessed by agency personnel using audit materials.

Research Triangle Institute (RTI), under contract to the Quality Assurance Division of EPA's Atmospheric Research and Exposure Assessment Laboratory, has investigated two audit materials for delivery of test compounds that could be used in performance audits during hazardous waste trial burns. The first audit material is XAD-2 resin contained in glass cartridges that has been spiked with known amounts of several semivolatile organic compounds. This audit material is sent to the laboratory involved in the trial burn. The laboratory recovers these compounds by Soxhlet extraction, concentrates the extract by Kuderna-Danish evaporation, and analyzes the extract by GC/MS.[1] This audit material is intended to assess only the analytical portion of Method 0010. The second audit material is an adsorbent, Carbotrap, contained in stainless steel cartridges that have been spiked with known amounts of semivolatile organic compounds and delivered to the trial burn sites. Once at the site, the compounds are thermally desorbed from the audit cartridge onto an operating Method 0010 sampling train, which collects them on XAD-2 resin for subsequent recovery and analysis. This audit material is intended to assess the combined sampling and analytical portions of Method 0010.

Measurement errors can occur in either the sampling or the analytical portions of Method 0010. The use of two different, but identically loaded,

audit materials allows one to begin to locate the source of an observed measurement error. If the measurement error were in the sampling portion, one would expect to see accuracy or precision problems for the Carbotrap audit cartridges, but not the XAD-2 audit cartridges. If the measurement error were in the analytical portion, one would expect to see accuracy or precision problems in both sets of audit cartridges. Statistical tests should be used to verify that any apparent accuracy or precision problems are significant. These audit materials cannot detect measurement errors arising from other sources such as the volumetric sampling rate.

An interlaboratory study was designed to quantitate the accuracy and precision of the loading, extraction, and analysis of compounds on both types of audit materials. The objective was to estimate the feasibility for using these materials in performance audits. Six compounds were studied on the two types of audit material: pyridine, toluene, o-xylene, chlorobenzene, 1,1,2,2-tetrachloroethane, and nitrobenzene.

In the sections that follow, the development of the Carbotrap and XAD-2 resin audit materials is summarized. The results of earlier RTI studies involving these materials are discussed. Results are reported for recovery and stability studies that were conducted prior to the interlaboratory study. Also, the results of the interlaboratory study are presented. Specifically, tables with average measured loadings, average bias percentages, between-laboratory variabilities and within-laboratory variabilities from the interlaboratory study are given in the Results and Discussion section.

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Two new audit materials have been developed to assess the accuracy and precision for both sampling and analysis portions of Method 0010. The first audit material, which employs XAD-2 resin, is used to evaluate the laboratory's ability to recover and analyze semivolatile organic compounds using Method 0010. Earlier RTI studies using XAD-2 resin showed that test compounds can be loaded by RTI and quantitatively recovered (i.e., 88 to 98 percent) by a cooperating laboratory from the resin. XAD-2 cartridges are stable under refrigeration over an eight-month period. The second audit material, which employs Carbotrap, is used to evaluate the laboratory's ability to sample, recover, and analyze for semivolatile organic compounds using Method 0010. Initial RTI experiments with Carbotrap audit cartridges showed that test compounds can be loaded onto the cartridges, thermally desorbed onto XAD-2 resin, and quantitatively recovered (i.e., 80 to 108 percent) from the resin. Results of desorption and analysis of spiked Carbotrap audit cartridges stored at room temperature showed that five of six test compounds are retained over a two-month period. Nitrobenzene does not appear to be stable on Carbotrap audit cartridges.

The two audit materials were also tested by five cooperating laboratories participating in an interlaboratory study. The overall mean bias percentage for the interlaboratory study was -29 percent. The mean bias percentages for individual laboratories ranged from -5 to -60 percent. This large range may be attributed as much to the particular test compounds chosen for the study as to the analytical capabilities of the cooperating laboratories. Most of the cooperating laboratories indicated that their Method 0010 analytical procedures had to be modified to measure these test compounds because of their low boiling points.

The between-laboratories variabilities (i.e., the relative standard deviations of mean measured loadings for the five laboratories) ranged from 10 to 68 percent with a mean value of 42 percent. The within-laboratory variabilities (i.e., the relative standard deviations of triplicate measurements within each laboratory) ranged from 0 to 94 percent with a mean value of 22 percent. Mean within-laboratory variabilities for individual laboratories ranged from 8 to 49 percent. These between-laboratories and within-laboratory variabilities generally agree with corresponding values obtained in an interlaboratory comparison study of methods for volatile and semivolatile compounds that was sponsored by EPA-Las Vegas.[9]

The XAD-2 and Carbotrap audit materials have been studied with sufficient detail to conclude that they are ready to be used as routine quality assurance audit materials. In general, test compounds can be loaded onto and recovered quantitatively from these audit materials. Additional work will be needed to study the stability of specific test compounds and to study the use of these audit materials for test compounds with higher boiling points than those of this study.

The results of the interlaboratory study indicate that these audit materials can successfully determine the precision and accuracy of Method 0010. The general agreement between the results of this study and the EPA-Las Vegas study support this conclusion. The precision and accuracy of the method can be better defined if the interlaboratory study would be extended to additional laboratories and to additional test compounds.

It is recommended that EPA consider establishing precision and accuracy specifications for Method 0010 and consider using these audit materials on a routine basis. Information derived by using these audit materials is an important component in assessing the performance of the emission test method, but should not be the sole criteria for acceptance of the emission test data.

SECTION 3

EXPERIMENTAL PROCEDURES

XAD-2 AUDIT MATERIAL FOR THE ANALYTICAL PORTION OF METHOD 0010

RTI performed initial studies of loading, desorbing, and analyzing test compounds on two types of audit material. The first type of audit material investigated was XAD-2 resin, which is a commercially available, pre-cleaned form of a polystyrene divinylbenzene porous polymer. This audit material is intended for use in assessing the analytical portion of Method 0010, which specifies XAD-2 as the sampling medium.

RTI has successfully employed XAD-2 resin as an intermediate-stage quality control material for three earlier studies involving sample collection of organic compounds by using the Method 0010.[2-6] Glass cartridges similar in design to the XAD-resin trap described in EPA Method 0010, were filled with 30 grams of XAD-2 resin.[1] Spike solutions of 5-μL volume containing one or more test compounds in methylene chloride were volatilized by a flash evaporating unit (see Figure 1). This unit is a modified gas chromatograph injection port maintained at 275 °C by a heat tape. Once volatilized, the test compounds were carried from the flash evaporator by a nitrogen flow and were mixed with room air being pulled through the XAD-2 audit cartridge at 1 cubic foot per minute (ft^3/min) by a Nutech Model 201 sample pump. The glass cartridge was immediately sealed after loading and was then stored under refrigeration.

To ensure that the test compounds were loaded in the correct amount, some of the spiked cartridges were measured by RTI prior to delivery of the remainder of the batch to a cooperating laboratory. The measured loadings were then compared to the loadings that were calculated from the spike solution injections onto the XAD-2 resin. The analytical techniques used by RTI included Soxhlet extraction of the XAD-2 resin for 16 hours with

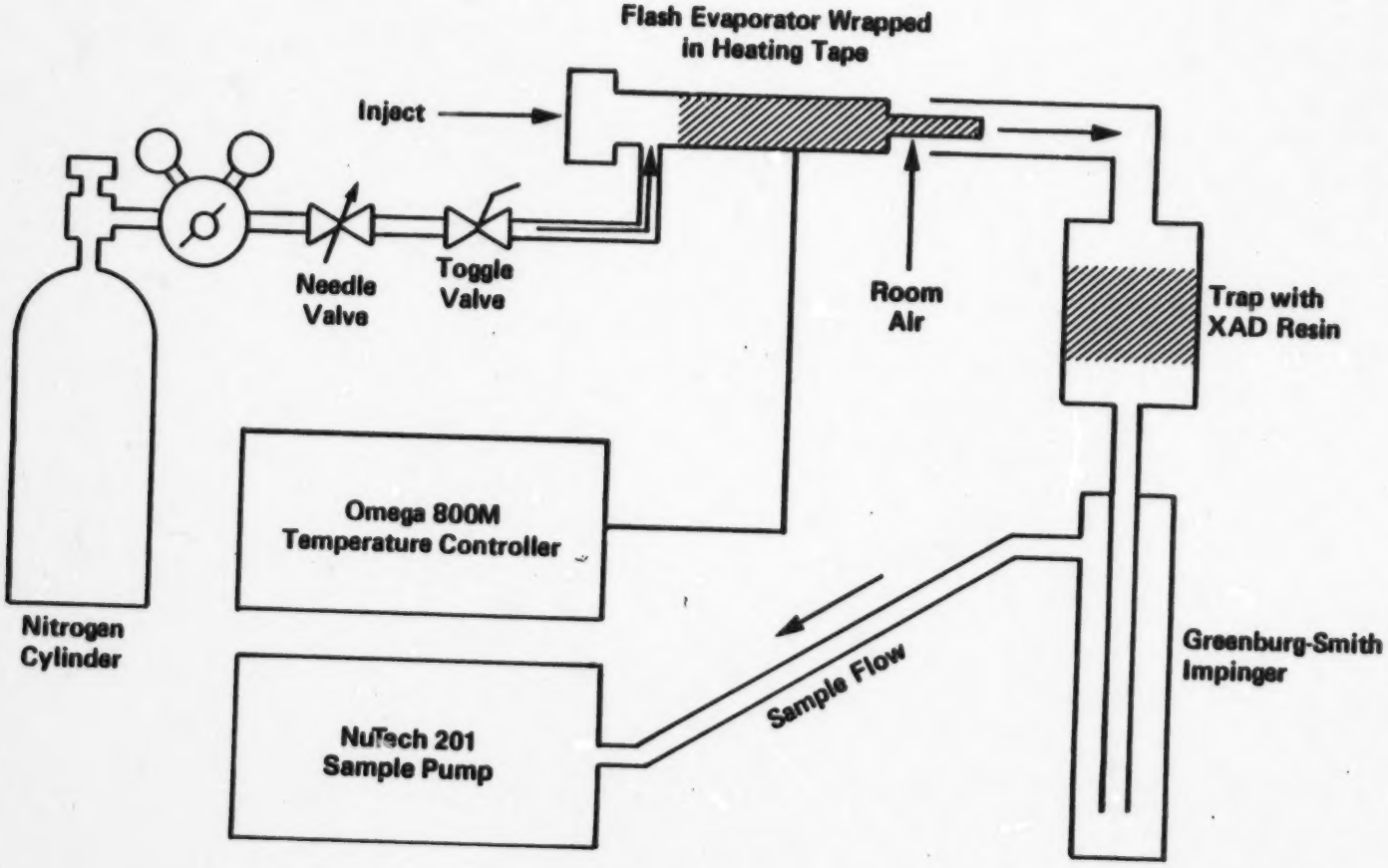

Figure 1. RTI laboratory apparatus used for loading organic compounds onto XAD-2 resin audit cartridges.

methylene chloride, which was followed by concentration of the extract by Kuderna-Danish evaporation. The concentrated extract was then diluted to a final volume and was analyzed by gas chromatography and flame ionization detection (GC/FID).

In these earlier studies, the cooperating laboratory analyzed the remaining spiked XAD-2 cartridges. The cooperating laboratory's analytical techniques included Soxhlet extraction of the XAD-2 resin with methylene chloride, which was followed by concentration of the extract by Kuderna-Danish evaporation. Extraction and evaporation losses were corrected for by the addition of deuterated versions of the test compounds to the XAD-2 resin to act as internal standards. The concentrated extract was analyzed by gas chromatography with mass spectroscopic detection (GC/MS). The cooperating laboratory was informed of the identity of the test compounds and, in one study, the loading levels of the test compounds.

CARBOTRAP AUDIT MATERIAL FOR THE SAMPLING AND ANALYTICAL PORTIONS OF METHOD 0010

The second type of audit material that RTI investigated is Carbotrap, which is a commercially-available graphitized carbon black. This audit material is intended for use in assessing the sampling and analytical portions of Method 0010. The audit cartridges were constructed from type 316 stainless steel tubing of 0.375-in. O.D. and 12-in. length. Approximately 0.8 g of Carbotrap was held centered in the cartridge by two quartz wool plugs. This arrangement produced an adsorbent bed length of about 2 in.

RTI investigated the recovery efficiencies and the stabilities of several test compounds on Carbotrap audit cartridges in an earlier study.[7] The recovery efficiencies were measured by direct thermal desorption of the test compounds from the cartridges onto a gas chromatograph. The cartridges were conditioned by purging them with nitrogen while heating them to 475 °C. After conditioning, the cartridges were allowed to cool to room temperature. Five microliters of a spike solution containing 1,4-dioxane, chlorobenzene, 1,1,2,2-tetrachloroethane , nitrobenzene, and tridecane in methylene chloride were volatilized by the flash evaporating unit, which was maintained at 275 °C (see Figure 2). The compounds were then carried

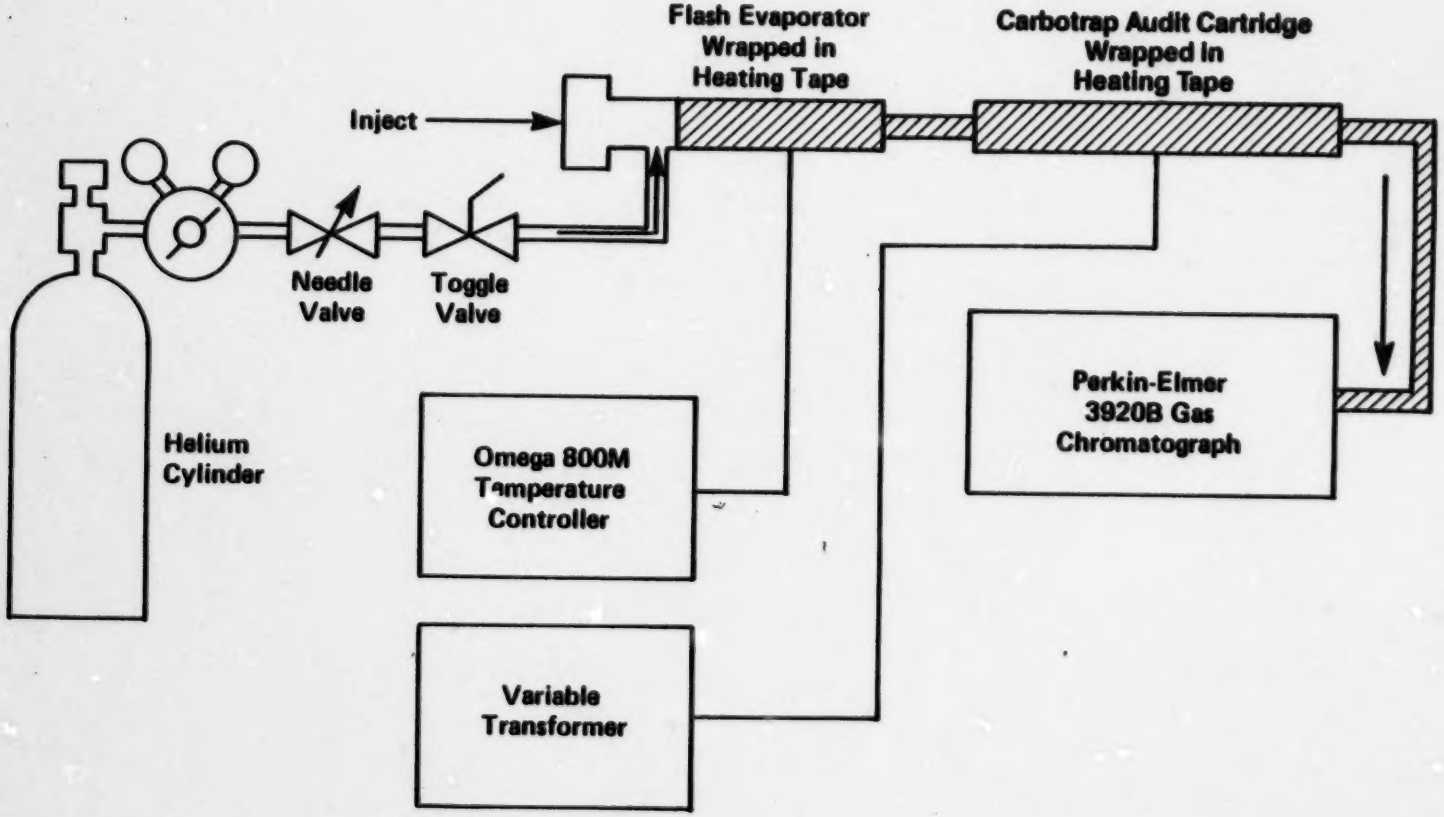

Figure 2. RTI laboratory apparatus used for Carbotrap audit cartridge recovery and stability studies.

by a helium flow and were trapped on the Carbotrap audit cartridge contained in an unheated tube furnace. The audit cartridges were analyzed by reversing the cartridge end-for-end and thermally desorbing the organics directly to a Perkin-Elmer Model 3920B gas chromatograph equipped with a flame-ionization detector.

Additional experiments which more closely simulate Method 0010 sampling procedures in the field were performed at RTI. The audit cartridges were conditioned and loaded with the same test compounds as were used for the direct thermal desorption tests. Once loaded, the cartridge was reversed, and the test compounds were thermally desorbed from the same end of the cartridge through which they were adsorbed. The desorbed test compounds were mixed with room air being pulled through an XAD-2 resin trap at 1 ft^3/min. The test compounds were recovered from the XAD-2 resin by Soxhlet extraction with methylene chloride. Internal standards were added to these Soxhlet extracts to quantify the evaporative losses. The extract was concentrated to 10 mL by a Kuderna-Danish evaporator. Subsequent analysis of the extract was performed by GC/FID.

AUDIT TECHNIQUES AND DESORPTION PROCEDURES

XAD-2 Audit Cartridges

The XAD-2 audit cartridge was designed to assess the cooperating laboratory's ability to quantitatively recover and analyze adsorbed organic compounds from the resin by solvent extraction. In most cases, the cooperating laboratory analyzed the audit cartridges alongside XAD-2 resin samples collected in the field by Method 0010. The analytical method described in EPA Method 0010[1] was also used for recovering the organic compounds. This method involved the extraction of the resin with methylene chloride. The volume of extract was reduced by Kuderna-Danish evaporation. The final volume of the concentrated extract was adjusted to adhere to instrumental detection limits.

Carbotrap Audit Cartridges

The Carbotrap audit cartridge was designed to assess the cooperating laboratory's ability to quantitatively sample, recover, and analyze test

compounds. The audit cartridge was connected directly to the sample probe of an operating Method 0010 sampling train (see Figure 3). The audit cartridge was the source of the organic compounds rather than the trial burn stack. Test compounds were thermally desorbed from the audit cartridge by purging it with nitrogen and heating it to 400 °C. The nitrogen containing the test compounds was mixed with filtered ambient air and was drawn into the sampling train. The test compounds were trapped on the XAD-2 resin, which was then handled as previously described.

INTERLABORATORY TESTING

The accuracy and precision of Method 0010 were measured by conducting an interlaboratory study involving cooperating laboratories. Audit samples were prepared and delivered to laboratory personnel who were experienced in Method 0010. The accuracy of the method was estimated by comparing the theoretical amount of test compounds loaded onto the audit cartridges to the amount measured by the cooperating laboratories. The average bias percentage between the theoretical loading and the multiple measured loadings for each laboratory's measurements of a compound was used as an index of the accuracy of the method. The between-laboratory variability was estimated by comparing the results of measurements at several different laboratories. The relative standard deviation of the average measured loadings for multiple laboratories' measurement of a compound was used as an index of the between-laboratory variability of the method. The within-laboratory variability was measured by providing several cartridges loaded at, or near, the same level. The relative standard deviation of the multiple measured loadings for each laboratory's measurements of a test compound was used as an index of the within-laboratory variability of the method.

Five laboratories agreed to participate in the interlaboratory study. Each laboratory received an audit kit containing seven Carbotrap audit cartridges, seven XAD-2 audit cartridges, and necessary supplies for thermal desorption of test compounds. The following test compounds were spiked onto both types of audit material: pyridine, toluene, o-xylene, chlorobenzene, 1,1,2,2-tetrachloroethane, and nitrobenzene. Of the seven Carbotrap audit cartridges supplied, three identical cartridges were loaded at approximately 200 μg of each compound per cartridge, three identical cart-

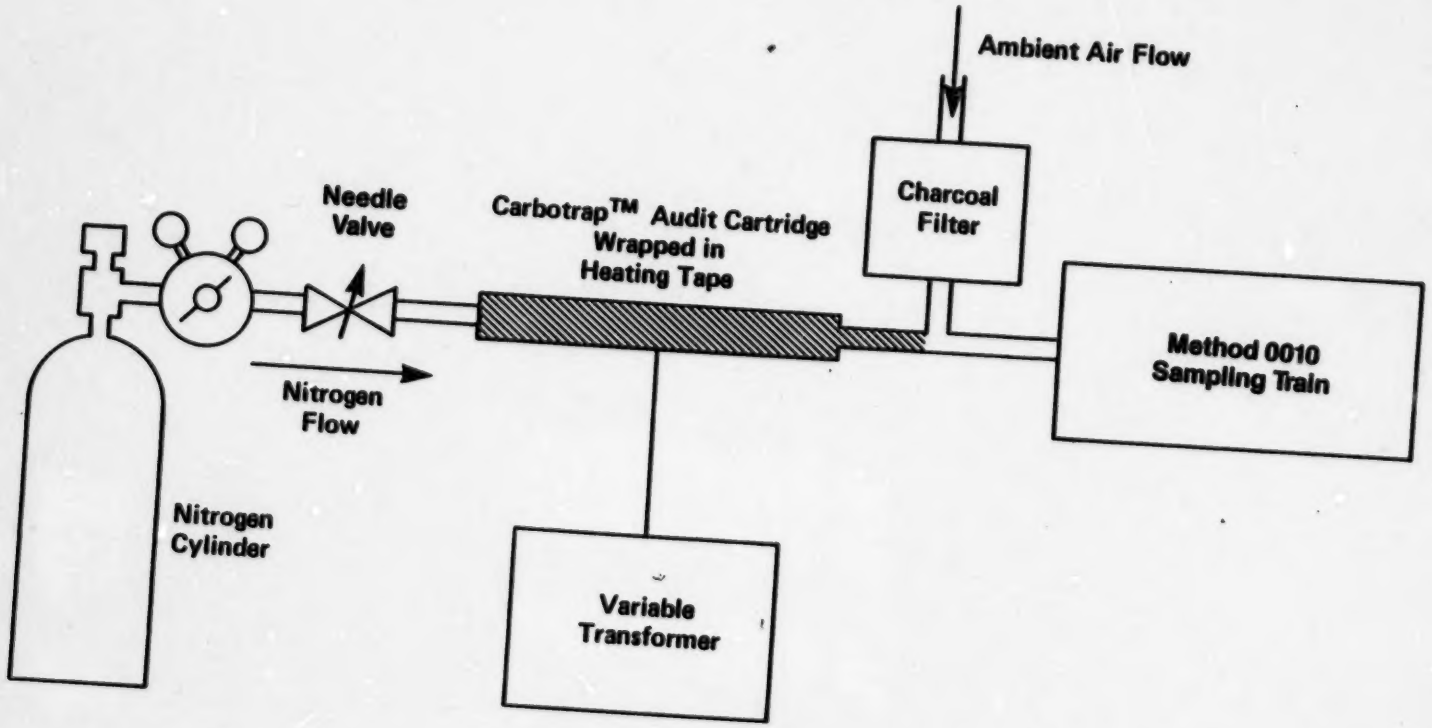

Figure 3. Apparatus used for desorbing Carbotrap audit cartridges during performance audits at hazardous waste trial burns.

ridges were loaded at approximately 2000 μg of each compound per cartridge, and one cartridge was a blank. The same loading scheme was also used for the seven XAD-2 audit cartridges.

The level at which each test compound was loaded onto the audit material was verified at RTI before the materials were shipped to the cooperating laboratories. Test compound names and a broad loading range (i.e., 50 to 5000 μg) were provided to the laboratories, but the exact level at which the compounds were loaded was not disclosed.

SECTION 4

RESULTS AND DISCUSSION

RECOVERY STUDIES FOR XAD-2 AUDIT CARTRIDGES

Earlier studies by RTI demonstrated that spiked XAD-2 resin can be used as an audit material for the analytical portion of Method 0010.[2-6] Table 1 gives mean recovery percentages and relative standard deviations for spiked XAD-2 cartridges that were prepared and analyzed by RTI. Table 2 gives the corresponding data for XAD-2 spiked cartridges that were prepared by RTI and analyzed by a cooperating laboratory. For the RTI's analyses, the uncorrected mean recovery percentages ranged from 73 to 103 percent and the corrected mean recovery percentages ranged from 79 to 140 percent. The uncorrected data indicate generally good recoveries for the test compounds. The corrected data were obtained by dividing the uncorrected mean recovery percentages of the test compounds by the mean recovery percentages of the internal standards. In general, the corrections improved the recovery percentages but in some cases the recovery percentages declined after correction. The corrected data suggest that it is difficult to correct for evaporative losses in test compounds with low boiling points. This difficulty is due to the use of internal standards with functional groups, boiling points, and recovery percentages that are different from those of the test compounds.

For the analyses by the cooperating laboratory, the corrected mean recovery percentages ranged from 88 to 98 percent. The improvement relative to RTI's corrected values is probably due to the cooperating laboratory's use of internal standards that are deuterated versions of the test compounds.

RECOVERY STUDIES FOR CARBOTRAP AUDIT CARTRIDGES

Another earlier study by RTI demonstrated that test compounds could be loaded and recovered successfully from spiked Carbotrap cartridges.[7] Table 3 presents the expected loadings and RTI's average recovery percentages for the

TABLE 1. Mean Recovery Percentages and Relative Standard Deviations for Spiked XAD-2 Cartridges Prepared and Analyzed by RTI

Year	Test Compound	Uncorrected		Corrected[a]	
		Mean % Recovery	RSD %	Mean % Recovery	RSD %
1984	Toluene	84.7	7.1	103.2	5.4
	Chlorobenzene	82.1	17.8	92.5	12.0
	1,1,2,2-Tetrachloroethane	72.7	40.8	79.3	41.2
	Decane	86.2	8.9	90.5	7.8
	Nitrobenzene	106.8[b]	3.8	110.8[b]	2.6
	Tridecane	85.1	9.1	84.3	7.5
	Pentadecane	87.3	7.8	88.0	6.0
1985	Toluene	89.0	1.1	115.7	8.0
	Chlorobenzene	92.5	1.3	113.9	5.4
	1,1,2,2-Tetrachloroethane	92.0	3.7	104.5	1.9
1986	Toluene	87.7	10.3	140.3	14.7
	Pyridine	84.8	1.0	133.6	3.1
	Chlorobenzene	99.1	2.4	114.2	8.9
	o-Xylene	102.7	3.6	108.2	5.0
	1,1,2,2-Tetrachloroethane	88.0	6.5	94.7	2.0
	Dodecane	97.9	5.2	--	--

[a]These recovery percentages are corrected by the recovery percentages of internal standard that were added to the Soxhlet extract.

[b]This recovery percentage was not corrected for a coeluting contaminant peak.

TABLE 2. Mean Recovery Percentages and Relative Standard Deviations for Spiked XAD-2 Cartridges Prepared by RTI and Analyzed by a Cooperating Laboratory

Year	Test Compound	Uncorrected		Corrected[a]	
		Mean % Recovery	RSD %	Mean % Recovery	RSD %
1985	Toluene	69.5	0.7	87.9	0.8
	Chlorobenzene	75.7	1.3	88.6	0.8
	1,1,2,2-Tetrachloroethane	77.3	0.2	--	--
1986[b]	Toluene	--	--	96.0	1.9
	Pyridine	--	--	98.0	1.0
	Chlorobenzene	--	--	98.1	1.7
	o-Xylene	--	--	93.4	0.7
	1,1,2,2-Tetrachloroethane	--	--	91.1	1.4

[a]These recovery percentages are corrected by the recovery percentages of deuterated test compounds added to the XAD-2 resin as internal standards.

[b]The loading levels of the test compounds were known by the cooperating laboratory.

TABLE 3. Results of Direct Thermal Desorption of Test Compounds From Spiked Carbotrap Cartridges Prepared and Analyzed by RTI

Compound	Amount loaded (μg/cartridge)	Average percent[a] recovery	Relative standard deviation (%)
1,4-Dioxane	206	98.7	8.1
Toluene	216	93.6	3.0
Octane	210	94.2	1.4
Chlorobenzene	220	96.5	2.8
1,1,2,2-Tetrachloroethane	238	132[b]	3.9
Nitrobenzene	240	56.7[b]	17.4
Dodecane	186	96.4	2.8
Tridecane	188	95.8	8.9
Tetradecane	190	100.7	0.4
Hexadecane	192	99.5	2.6

[a]Average recovery for three audit cartridges

[b]Recoveries probably due to poor chromatographic separation of these test compounds that is associated with with direct thermal desorption. Better recoveries for these compounds were obtained by thermal desorption onto XAD-2 resin followed by liquid injection of the extract into the gas chromatograph.

TABLE 4. Results of Thermal Desorption of Test Compounds from Spiked Carbotrap Cartridges onto XAD-2 Resin Prepared and Analyzed by RTI

Compound	Amount loaded (μg/cartridge)	Average percent[a] Recovery	Relative standard Deviation, (%)
1,4-Dioxane	205	90.7	5.2
Chlorobenzene	220	102	3.3
1,1,2,2-Tetrachloroethane	317	104	3.7
Nitrobenzene	239	108	2.8
Tridecane	226	96.4	10.4
Pentadecane	229	79.5	2.8

[a]Average recovery for three audit cartridges.

direct thermal desorption technique used to analyze the Carbotrap audit cartridges. The average recovery percentages ranged from 57 to 132 percent. Better than 90-percent recovery percentages are noted for toluene, chlorobenzene, and other test compounds. The high recovery for 1,1,2,2-tetrachloroethane and low recovery for nitrobenzene are probably due to poor chromatographic separation of these test compounds that is associated with direct thermal desorption.

The recovery percentages are calculated by the following method. An empty cartridge is placed in the thermal desorption unit, replacing the audit cartridge. The temperature of the empty cartridge is maintained at 275 °C by a heat tape. The same amount of test compounds is injected into and passes through the empty cartridge to the gas chromatograph as is loaded on and desorbed from the audit cartridges. With this method, recovery efficiencies can be compared on a one-to-one basis. Blank Carbotrap cartridges were also desorbed; the chromatographic results show no compound elution that match the retention times of the target compounds.

The earlier RTI results obtained from thermal desorption of test compounds from Carbotrap audit cartridges onto XAD-2 resin are shown in Table 4. These data indicate that 1,1,2,2-tetrachloroethane, chlorobenzene, nitrobenzene, and other test compounds can be loaded and recovered quantitatively on this audit material. The average recovery percentages ranged from 80 to 108 percent. The recovery percentages for 1,1,2,2-tetrachloroethane and nitrobenzene were close to 100 percent which is much better than the recovery percentages observed for direct thermal desorption. The reason for the poor recovery percentage of pentadecane is unknown. This difference in recovery percentages is probably due to the method used to deliver the test compounds for chromatographic separation. A liquid injection of the XAD-2 extract delivers the recovered test compounds to the gas chromatograph as compared to a flow of gas used with direct thermal desorption. A blank Carbotrap cartridge was also desorbed onto the XAD-2 resin. The only peaks that could be seen in the chromatogram of the blank extract either were methylene chloride contaminants/preservatives or were peaks whose area counts were below the minimum area cutoff of the integrator.

STABILITY STUDIES FOR CARBOTRAP AUDIT CARTRIDGES

The stability of test compounds on Carbotrap audit cartridges was studied over a two-month period. Table 5 presents the test compounds studied, the levels at which the compounds were loaded, and the recovery percentages at three time intervals. The direct thermal desorption technique was used to analyze the stability cartridges. Improved chromatographic separation of 1,1,2,2-tetrachloroethane and nitrobenzene, as compared to the recovery study using direct thermal desorption, was achieved by changing instrumental parameters. Average recovery percentages ranged from 91 to 99 percent after one day, from 81 to 100 percent after thirty days and from 76 to 95 percent after sixty days. These data indicate that pyridine, 1,1,2,2-tetrachloroethane, toluene, chlorobenzene, and o-xylene can be loaded and recovered quantitatively on this audit material. Recovery efficiencies indicate five of the six test compounds showed little change ($\pm$ 7 percent) in the amount loaded when stored at room temperatures for 60 days. The recovery efficiency of nitrobenzene is lower than other compounds through 60 days. These data and data from earlier RTI studies suggest that nitrobenzene is not stable on Carbotrap audit cartridges.[7]

STABILITY STUDIES FOR XAD-2 AUDIT CARTRIDGES

Similar stability studies were performed with the same six compounds on XAD-2 resin. The test compounds were spiked directly onto the XAD-2 resin contained in the audit cartridges. The cartridges were stored under refrigeration. Excellent recoveries of test compounds through 269 days were obtained as indicated in Table 6. Average recovery percentages ranged from 98 to 106 percent after 4 days, from 99 to 106 percent after 151 days and from 97 to 109 percent after 269 days. RTI did not use internal standards for these analyses. The excellent recovery percentages during the stability studies for XAD-2 audit cartridges are due to improvements that were made in the Kuderna-Danish evaporator after the completion of the recovery studies. Nitrobenzene is stable on XAD-2 audit cartridges.

INTERLABORATORY TESTING

After a batch of audit cartridges had been loaded with the test compounds, a portion of the batch was analyzed by RTI. The purpose of the

TABLE 5. Results of RTI Carbotrap Stability Study Using Direct Thermal Desorption

Test Compound	Amount loaded (μg)	Average percent recovery[a] after 1 day	30 days	(% change)[b]	60 days	(% change)[c]
Pyridine	197	92.8	93.2	(+0.43)	92.1	(-0.75)
1,1,2,2-Tetrachloroethane	319	91.1	90.4	(-0.77)	85.4	(-6.3)
Toluene	174	94.8	97.7	(+3.1)	90.3	(-4.7)
Chlorobenzene	221	94.1	95.2	(+1.2)	89.1	(-5.3)
o-Xylene	176	98.6	99.7	(+1.1)	95.0	(-3.7)
Nitrobenzene	241	98.8	81.1	(-18.0)	76.1	(-23)

[a]Average recovery for three stability cartridges.

[b]Percent change = $\frac{\text{30 days\% - 1 day\%}}{\text{1 day\%}}$

[c]Percent change = $\frac{\text{60 days\% - 1 day \%}}{\text{1 day\%}}$

TABLE 6. Results of RTI XAD-2 Resin Stability Study

Test Compound	Amount loaded (μg)	Average percent recovery[a] after 4 days	151 days	(% change)[b]	269 days	(% change)[c]
Pyridine	197	105	99.3	(-5.4)	109	(+3.8)
1,1,2,2-Tetrachloroethane	319	98.4	104	(+5.7)	97.8	(-0.61)
Toluene	174	101	102	(+0.99)	96.7	(-4.3)
Chlorobenzene	221	106	106	(0.0)	101	(-4.7)
o-Xylene	176	104	102	(-1.9)	98.0	(-5.8)
Nitrobenzene	241	102	105	(+2.9)	104	(+2.0)

[a]Average recovery for three stability cartridges.

[b]Percent change = $\frac{\text{151 days\% - 4 days\%}}{\text{4 days\%}}$

[c]Percent change = $\frac{\text{269 days\% - 4 days\%}}{\text{4 days\%}}$

analysis was to verify that the batch had been loaded properly. The remainder of the batch was sent to the cooperating laboratory.

Table 7 summarizes the mean recovery percentages and relative standard deviations for audit cartridges that were loaded and analyzed by RTI during the interlaboratory study. Because direct thermal desorption was used in the analysis of the Carbotrap audit cartridges, some of the test compounds' peaks were not completely resolved on the chromatogram. The unresolved peaks are treated as a single peak for the purpose of calculating the recovery percentage. In general, the mean recovery percentages ranged from 94 to 110 percent. The relative standard deviations ranged from 1 to 23 percent with a mean value of 6 percent.

Table 8 gives the test compounds loaded onto the audit cartridges, the expected loadings of each compound on the audit cartridges, and the average measured loadings for audit cartridges that were loaded by RTI and analyzed by the cooperating laboratories. The latter values are the averages for measurements of three identical cartridges that were given to the cooperating laboratories.

Note that one of the three measured loadings for the Lab A/o-xylene/XAD-2/high-loading combination has been altered from the value that Lab A reported. A review of copies of Lab A's chromatograms revealed a transcription error when the area counts under one o-xylene peak was transcribed. Lab A reported a value of 5931 micrograms, rather than 593 micrograms which is the correct value. The correct value is used for all calculations in this report because it will yield more realistic estimates of Lab A's analytical accuracy and precision.

The test compounds loaded, expected loadings, and the average bias percentage between average measured loadings and expected loadings from the five cooperating laboratories are shown in Table 9. The overall average bias percentage for both audit materials in the interlaboratory study is -27 percent. The unusually high value for the Lab E/pyridine/XAD-2/low-loading combination was found to be an outlier at the 99-percent confidence level, relative to the other measured pyridine loadings.[8] If this value is discarded, the overall bias percentage is -29 percent. The overall average bias percentage for the Carbotrap audit cartridges alone is approximately equal to the corresponding value for the XAD-2 audit cartridges alone

TABLE 7. Mean Recovery Percentages and Relative Standard Deviations for Audit Cartridges Prepared and Analyzed by RTI During the Interlaboratory Study.

Test Compound	Low-Loading Carbotrap Cartridges			High-Loading Carbotrap Cartridges		
	Number of Values	Mean % Recovery	RSD (%)	Number of Values	Mean % Recovery	RSD (%)
Pyridine	4	97.1	5.6	4	101.7[b]	1.3[b]
Toluene	4	102.6	13.7			
o-Xylene	4	105.4[a]	14.6[a]	4	101.5[a]	1.6[a]
1,1,2,2-Tetrachloroethane						
Chlorobenzene	4	103.6	12.7	4	101.6	2.2
Nitrobenzene	4	100.4	22.6	4	101.2	3.7

Test Compound	Low-Loading XAD-2 Cartridges			High-Loading XAD-2 Cartridges		
	Number of Values	Mean % Recovery	RSD (%)	Number of Values	Mean % Recovery	RSD (%)
Pyridine	7	109.5	6.8	7	94.6	4.4
Toluene	7	104.3	3.9	7	94.2	5.9
o-Xylene	7	107.6	2.6	7	97.3	2.8
1,1,2,2-Tetrachloroethane	7	106.6	6.3	7	100.6	5.3
Chlorobenzene	7	105.3	3.7	7	96.6	3.8
Nitrobenzene	7	103.9	6.3	7	99.6	5.8

[a]The o-xylene and 1,1,2,2,-tetrachloroethane peaks were not completely resolved in the chromatograms as a result of direct thermal desorption.
[b]The pyridine and toluene peaks were not completely resovled in the chromatograms as a result of direct thermal desorption.

TABLE 8. Average Measured Loadings in the Interlaboratory Study

Carbotrap Audit Cartridge

Compound	Expected loading (μg/cartridge) Low	High	Cooperating laboratories and average measured loadings (μg) Lab A Low	Lab A High	Lab B Low	Lab B High	Lab C Low	Lab C High	Lab D Low[a]	Lab D High	Lab E Low[b]	Lab E High
Pyridine	197	1978	71	632	166	1367	287	2266	72	1144	196	1366
Toluene	174	1748	91	267	127	767	187	1967	367	718	135	1233
o-Xylene	178	1768	74	528	143	1667	177	2133	97	1029	135	1266
1,1,2,2-Tetra-chloroethane	319	3198	163	986	223	1833	323	3667	146	2343	236	1733
Chlorobenzene	221	2218	54	395	193	913	237	2433	168	978	256	1567
Nitrobenzene	241	2418	177	1313	266	1767	266	2566	267	3193	166	2567

XAD-2 Resin Audit Cartridge

Compound	Expected loading (μg/cartridge) Low	High	Cooperating laboratories and average measured loadings (μg) Lab A Low	Lab A High	Lab B Low	Lab B High	Lab C Low	Lab C High	Lab D Low	Lab D High	Lab E Low[c]	Lab E High
Pyridine	197	1978	94	794	157	1266	237	2166	114	715	866[d]	2133
Toluene	174	1748	75	293	85	597	147	1267	81	516	266	1333
o-Xylene	178	1768	82	692	123	947	153	1333	98	781	216	1333
1,1,2,2-Tetra-chloroethane	319	3198	152	1174	213	1733	223	2133	232	1668	366	2133
Chlorobenzene	221	2218	64	661	183	726	187	1667	185	957	366	2167
Nitrobenzene	241	2418	151	1343	197	1567	227	1933	218	2362	296	1866

[a]One of three Kuderna-Danish evaporators went dry; data for this sample are not included in the calculations.
[b]One of three loaded cartridges was reported as "none detected"; data for this sample are not included in the calculations.
[c]Two of three loaded cartridge were reported as "sample lost"; data for these samples are not included in the calculations.
[d]Found to be an outlier at the 99 percent confidence level.

TABLE 9. Average Bias Percentages for Measured Loadings in the Interlaboratory Study

Carbotrap Audit Cartridge

Compound	Expected Loading (μg/cartridge) Low	High	Cooperating laboratories and average bias percentage[a] Lab A Low	Lab A High	Lab B Low	Lab B High	Lab C Low	Lab C High	Lab D Low[b]	Lab D High	Lab E Low[c]	Lab E High
Pyridine	197	1970	-64	-68	-19	-31	+5	+12	-63	-42	- 4	-34
Toluene	174	1740	-48	-88	-27	-56	+7	+13	+76	-59	-22	-29
o-Xylene	176	1760	-58	-78	-19	-39	+1	+21	-45	-42	-23	-32
1,1,2,2-Tetra-chloroethane	319	3190	-49	-69	-38	-43	+1	- 4	-56	-27	-28	-46
Chlorobenzene	221	2210	-76	-82	-13	-59	+7	+10	-51	-56	+13	-29
Nitrobenzene	241	2410	-27	-46	-17	-27	-17	+ 4	-14	+32	-34	+ 7

XAD-2 Resin Audit Cartridge

Compound	Expected loading (μg/cartridge) Low	High	Cooperating laboratories and average bias percentage Lab A Low	Lab A High	Lab B Low	Lab B High	Lab C Low	Lab C High	Lab D Low	Lab D High	Lab E Low[d]	Lab E High
Pyridine	197	1970	-52	-68	-28	-39	+28	+ 7	-42	-64	+386[e]	+ 8
Toluene	174	1740	-57	-83	-51	-66	-16	-27	-53	-71	+ 15	-23
o-Xylene	176	1760	-53	-61	-38	-46	-13	-24	-44	-56	+ 19	-24
1,1,2,2-Tetra-chloroethane	319	3190	-52	-63	-33	-46	-38	-33	-27	-48	+ 13	-33
Chlorobenzene	221	2210	-71	-73	-53	-67	-15	-25	-52	-57	+ 36	- 2
Nitrobenzene	241	2410	-37	-44	-18	-35	- 6	-28	-18	- 2	+ 28	-25

[a]Average bias percentage = 100 [(average measured loading - expected loading)/expected loading].
[b]One of three Kuderna-Danish evaporators went dry; data for this sample are not included in the calculations.
[c]One of three loaded cartridges was reported as "none detected"; data for this sample are not included in the calculations.
[d]Two of three loaded cartridges were reported as "sample lost"; the average percent bias is for a single sample.
[e]Found to be an outlier at the 99 percent confidence level.

(i.e.,-28 and -32 percent, respectively and excluding the anomalous Lab E value). The overall average bias percentage for both types of low-loading audit cartridges (excluding the outlier) is -23 percent. This value is less than the corresponding value of -35 percent for both types of high-loading audit cartridges.

The overall average bias percentages for each laboratory are given below:

Lab	Overall Mean Bias Percentage
A	-60
B	-39
C	- 5
D	-39
E	-11[a]

[a]Excluding the outlier.

These values are the means of all of each laboratory's mean bias percentages that are given in Table 9.

The results from the interlaboratory study show large variations in bias percentages between laboratories and within individual laboratories. For example, the average bias percentage for the six test compounds at the two loadings for both types of audit material for Laboratory A is -60 percent (range of -88 to -27 percent) compared to Laboratory C's average bias percentage of -5 percent (range of -33 to +21 percent).

The large range in the average bias percentages for test compounds within a particular laboratory may be attributed to the group of test compounds selected for the interlaboratory study. The individual compounds were selected with more emphasis placed on the class of compounds which they represent rather than the boiling point of the particular compound. Many cooperating laboratories indicated the need to modify their existing Method 0010 analytical procedures to accommodate low-boiling test compounds such as toluene (B.P.= 110 °C), chlorobenzene (B.P.= 132 °C), and 1,1,2,2-tetrachloroethane (B.P.= 146 °C).

STATISTICAL ANALYSIS

The average measured loadings that are given in Table 8 were used to calculate the between-laboratory variability. This value is defined as the relative standard deviation of the average measured loading among the five

cooperating laboratories for each test compound/audit material/loading combination. The between-laboratory variabilities for the interlaboratory study are given in Table 10.

TABLE 10. Relative Standard Deviations for the Average Measured Loadings Between the Cooperating Laboratories in the Interlaboratory Study

Compound	Carbotrap audit cartridge		XAD-2 resin audit cartridge	
	Low	High	Low	High
Pyridine	47	42	43[a]	49
Toluene	49	68	46	59
o-Xylene	33	48	38	30
1,1,2,2-Tetrachloroethane	32	40	33	22
Chlorobenzene	51	61	63	56
Nitrobenzene	10	32	23	21

[a]The single measured loading from Lab E is excluded an an outlier.

These values range from 10 to 68 (excluding the outlier) with an overall average value of 42. In general, the between-laboratories variabilities are consistent across the various audit material/loading combinations and across the various test compounds. The mean relative standard deviation across the various audit material/loading combinations are given below:

Audit Material/ Loading Combination	Mean RSD %
Carbotrap/low loading	37
Carbotrap/high loading	48
XAD-2/low loading	41[a]
XAD-2/high loading	40

[a]Excluding the outlier

The mean relative standard deviations across the various test compounds are given below:

Test Compound	Mean RSD %
Pyridine	45[a]
Toluene	56
o-Xylene	37
1,1,2,2-Tetrachloroethane	32
Chlorobenzene	58
Nitrobenzene	22

[a]Excluding the outlier

These mean relative standard deviations do not include the unusually high value (i.e., 105 percent) for pyridine/XAD-2/low-loading combination. This value can be discarded as an outlier at the 99-percent confidence level.

The relative standard deviation for the three measured loadings within each laboratory are given in Table 11. These values are used to calculate the within-laboratory variabilities, which are defined as the mean relative standard deviation for each laboratory. These values ranged from 0 to 94 percent with an overall mean value of 22 percent. The minimum, maximum and mean relative standard deviations for each laboratory are given below:

	Relative Standard Deviation (%)		
Lab	Minimum	Maximum	Mean
A	9	94	49
B	2	20	8
C	0	31	11
D	7	54	23
E	0	82	19

In general, the relative standard deviations are consistent within each laboratory, but tend to differ between laboratories. Also, note that within-laboratory variabilities are less than the between-laboratories variability. The mean within-laboratory variability (i.e., 22 percent) is approximately one-half of the mean between-laboratories variability (i.e., 42 percent).

COMPARISON WITH OTHER RESEARCH

In 1984, EPA-Las Vegas published the results of an interlaboratory comparison study of methods for volatile and semivolatile compounds.[9] Nine

TABLE 11. Relative Standard Deviations for Measured Loadings Within Each Cooperating Laboratory in the Interlaboratory Study

Carbotrap Audit Cartridge

Compound	Cooperating laboratories and percent relative standard deviations									
	Lab A		Lab B		Lab C		Lab D		Lab E	
	Low	High	Low	High	Low	High	Low[a]	High	Low[b]	High
Pyridine	49	36	11	11	31	16	38	7	82	13
Toluene	62	47	12	11	28	8	36	25	37	5
o-Xylene	73	46	8	28	21	3	26	14	37	0
1,1,2,2-Tetrachloroethane	31	32	9	3	24	7	25	29	18	17
Chlorobenzene	66	28	13	18	28	6	29	27	28	21
Nitrobenzene	24	19	9	9	9	0	8	15	6	13

XAD-2 Resin Audit Cartridge

Compound	Cooperating laboratories and relative standard deviations									
	Lab A		Lab B		Lab C		Lab D		Lab E	
	Low	High	Low	High	Low	High	Low	High	Low[c]	High
Pyridine	92	22	4	8	6	8	7	14	--	11
Toluene	73	9	2	9	18	5	16	38	--	17
o-Xylene	94	17	5	8	8	9	16	24	--	17
1,1,2,2-Tetrachloroethane	89	17	5	3	7	18	28	13	--	11
Chlorobenzene	69	21	6	6	8	9	13	54	--	13
Nitrobenzene	68	13	8	4	11	11	38	21	--	6

[a]One of three Kuderna-Danish evaporators went dry; data for this sample are not included in the calculations.
[b]One of three loaded cartridges was reported as "none detected"; data for this sample are not included in the calculations.
[c]Two of three loaded cartridges were reported as "sample lost"; no relative standard deviation can be calculated.

laboratories participated in the study. Semivolatile compounds with boiling points up to 500 °C were spiked onto five different waste samples. Each laboratory was requested to analyze three replicates of each waste sample. The semivolatiles were extracted from the waste samples with methylene chloride by sonification under neutral anhydrous conditions. They were analyzed by a GC/MS with a fused silica capillary column. The high and low spike levels corresponded to those levels that would give approximately 50 and 10 nanograms of the semivolatile compounds on the GC column during analysis if 100-percent extraction recovery were achieved.

The relative standard deviation for the within-laboratory component was less than 30 percent and the between-laboratories variability was about twice the value. The ranges of both components were 5 to 300 percent. It appears that within-laboratory and between-laboratories variabilities from the current study generally agree with those from the EPA-Las Vegas study.

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